BY - PRODUCTS FROM WOOL - WASHING

BY

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ARMOUR INSTITUTE OF TECHNOLOGY

1916

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BY-PRODUCTS FROM WOOL WASHING

A Thesis

Presented by

SAMUEL BUTTERMAN

and

VICTOR E MARX

to the

PRESIDENT AND FACULTY

of the

ARMOUR INSTITUTE OF TECHNOLOGY

For the Degree of

BACHELOR OF SCIENCE IN CHEMICAL ENGINEERING

Having Completed the Prescribed

Course of Study

in

Chemical Engineering

1916

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ACKNOWLEDGMENT.

The authors desire, at the outset, to express their acknowledgment to Professor McCormack for valuable suggestions and assistance rendered during the course of the investigational work, and to express an indebtedness to the Boynton Wool Scouring Co., Chicago, for their courtesy in allowing the authors to visit their modern wool scouring plant.

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INTRODUCTION.

The consideration of the topic "Wool Washing and Recovery of By-Products" arises out of the three-fold circumstance that the ordinary method of preparing wool for manufacturing purposes by the process of scouring, yields a product with objectionable properties; secondly, that the by-products which are, in this country, for the most part being discarded to waste, have at the present time a high value; and lastly, there has been presented, as a means of obviating the first difficulty and profiting by the high return offered in the recovery of these valuable by-products, the possibility of the utilization of a coal-tar distillate, of little value otherwise, to be applied to a solvent process of purification.

THE INDUSTRY OF WOOL-WASHING.

Nature of Wool.

Wool fiber is the hairy covering (or fleece) of the sheep. It is a growth originating in the skin, springing from a root or hair follicle. In its natural state in the fleece, wool is contaminated with a number of impurities. These may be classified as follows:[1]

(a) Wool grease, which occurs in large quantities as an external coating on the fiber; it is a natural exudation of the sheep and serves as a protection to the fiber, preventing it from becoming felted and mechanically injured. It differs from other animal fats in that it does not consist of the glycerides of the fatty acids, and is very difficultly saponifiable with caustic alkalies. It cannot be saponified with aqueous potash, but is readily saponified by

[1] Roger's Industrial Chemistry.

heating at 100C in a closed vessel with alcoholic potash. The saponification number of lanolin determined in this manner is found to be 83.44, which is about one half that of ordinary fats.[2] Wool grease possesses more the chemical properties of a wax, as it is composed mostly of higher solid alcohols known as cholesterin and isocholesterin, both in the free state and as esters with the fatty acids. Though insoluble in water and not saponifiable by alkalies, cholesterin is easily emulsified, a property on which is based the usual method of wool scouring. Wool grease however, is easily soluble in naptha and other volatile solvents.

(b) Suint, or dried up perspiration, consisting largely of potash salts of organic acids and soluble in water. The presence of potassium is accounted for by the well known

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[2] Jr. London Chem. Soc. 1893 vol. 2.

fact that sheep draw from the land on which they graze, a considerable quantity of potash, which, after circulating in their blood, is excreted from the skin with the sweat in combination with which it is deposited in the wool.[3]

(c) Miscellaneous dirt, such as dust, sand, vegetable matter, tar, gravel, etc..

PREPARATION OF WOOL FOR MANUFACTURE.

Before the wool can be spun in the textile industries, it must be freed from grease and adhering impurities. For this purpose two methods are in use: A. Scouring Process, and B. Solvent Process.

A. Scouring Process.

The ordinary method of cleansing wool is by the scouring process in which the dirty greasy, wool is scoured in a warm soap solution

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3. J.L.Smith, Chem. News Vol. 23 p 9, Jan 1871

to which more or less soda ash is added. The temperature of scouring should not be above 140F else the fiber will be injured by the action of the alkali. The wool grease is easily emulsified by the alkalies, whereas the suint is dissolved by the water, the other impurities being removed mechanically by the action of the water. After scouring in the soap solution the wool is thoroughly rinsed in warm water, and finally squeezed and dried.

The potash salts existing in the wool suint and removed by the scouring are valuable by-products which are capable of recovery. In many European plants these salts as well as the grease are recovered and utilized, in this country however, the wash waters containing the suint are run to waste. The amount of grease and dirt in wool varies greatly with the breed and cultivation of the sheep. The finer stapled wools usually contain the largest proportion of grease. The amount of dirt and

vegetable matter will depend largely on the character of the range on which the sheep are grown; a dry sandy soil usually producing a large amount of dust in the wool. The amount of loss caused in scouring wool is known as its shrinkage and this amounts to 40-70% on the weight of the raw fleece.

The scouring of wool is usually conducted by machinery with the object of agitating the fiber the least possible degree so as to avoid felting. The machines in general use are long tanks arranged in tandem order. The greasy wool is introduced into the first tank by means of a travelling apron and is slowly carried through the scouring liquor by moving forks until it is caught up by squeeze rolls passed into the second tank, where it is carried forward in the same manner through another soap solution; it is finally carried through a third tank containing fresh warm water for rinsing. The scouring liquor is introduced fresh into

the second tank and passes thence into the first tank, so as to have the clean wool coming from the fresh liquor. In some places the spent scouring liquors are worked up to recover the potash salts by simple evaporation and calcination, when from 5 to 10% (on the weight of the raw wool) potash may be obtained. Again, the spent liquors are sometimes treated with sulphuric acid in order to decompose the soaps and liberate the fatty matter. The latter, together with the associated wool grease, are settled out on the surface of the liquor from which they are removed. In some instances the scouring liquors are centrifuged to recover the crude wool fat.

The objectionable feature of this treatment is that the alkali has a destructive effect upon the wool causing it to turn yellow; accordingly there is becoming of great importance in this country another form of scouring known as the solvent process, which

leaves the fiber in a much better condition and the recovered grease is of sufficient value to pay for the cost of scouring.

B. The Solvent Process.

The literature contains many accounts of various solvent processes in which the main feature has been to leach the grease from the wool with a volatile solvent, such as benzol, carbon disulphide, carbon tetrachloride, petroleum benzine, etc., and subsequently subject the solution to distillation, recovering the solvent for further use, while the grease remained behind as a residue in the still. The application of this process on an industrial scale has in most instances failed to survive beyond the experimental stage and there are at present but few plants in the world which are operating. The main difficulty, practically, has arisen in connection with the handling of the volatile solvents,

which entails a large loss of an expensive raw material, and presents further, a fire hazard.

UTILIZATION OF THE BY-PRODUCTS.

Wool fat mixed with water, in relation to medicine, has first been mentioned by Dioscorides, a Greek physician and author, living in the first century A.D.. Plinius in his writings, gives an elaborate method for the extraction of wool grease from wool in which he calls his product "oesypus". In 1882, Drs. Braun and Liebrich took out a patent for the purification of wool fat, which, mixed with water they called "lanoline". They described their product as "a mixture of pure wool fat with water".[4] The appearance of the preparation showed it to be far from pure; the color of the anhydrous wool fat was greenish brown and when mixed with water it assumed a dirty yellow color which turned brown on standing.

4. Jr. Soc. Chem. Ind. Vol. 9,1890 p 356.

Lanolin has now become firmly established in the Pharmacopoeia because of its great therapeutic value. It is already taking the place of vaseline, paraffin, and lard. Its efficiency in the composition of plasters, salves, and ointments has already been established beyond doubt, and its superiority is due to the extraordinary readiness with which it is absorbed by the skin, to a degree not known to belong to any other fatty substance. Hence its utility for introducing medical preparations into the system through the skin.

Besides the great number of medical uses which have been found for lanolin during the short period of its existence, it has already been introduced into various branches of industry, such as perfumery, soaps, creams and pomades, also for greasing leather, for belting and for improving the pliability of leather.[5]

[5] Jr.Soc.Chem.Ind. 1885 p 579.

The present market quotation (wholesale price) for lanolin as taken from the Oil, Paint and Drug Reporter for May 15,1916 page 48, is:

Hydrous Lanoline, per lb.----$1.05

Anhydrous Lanoline, per lb.---$1.45

With regard to potassium, it need only be mentioned that its wide application and present high value make its recovery a very desirable end.

THE PROBLEM.

The problem as here considered by the authors was to examine a particular coal-tar distillate with respect to its applicability to a solvent process, and to investigate the nature of the resulting products.

THE INVESTIGATION.

Raw Materials.

Nature of the Solvent.

The solvent as furnished by the Chattanooga Gas and Coal Products Co., of Chattanooga, Tennessee, was a coal-tar fraction from the plant stills, distilling within the range 200 to 250 C. It was a clear, dark yellow, mobile, oily, liquid of specific gravity 0.912 at 20C, having a pungent odor and burning with a very smoky flame. Chemically, the liquid is known to contain homologues of benzene such as xylene, cymene, etc., which, however are too difficult to separate because they are present in such small quantities. Hence this operation is not carried on in this country and the fraction is of comparatively little value beyond its application as a solvent for paints, and for cleaning clothes.

The Wool.

The wool employed was secured from the packing house of Armour and Co., and is known as their A A Wool, No.52, Armour. It was essentially a crude wool with the normal content of grease and other concomitant impurities.

PRELIMINARY EXPERIMENTATION.

It was desired at the outset to make some preliminary observations along the lines which it was proposed to follow in the main operations, in order to anticipate probable sources of difficulty in the subsequent investigation, and accordingly, with this end in view, 190cc of crude solvent (the amount then on hand) was run in upon 10 grams of raw wool, and the leaching allowed to extend over 3 days by mere contact. At the end of this time the liquor was drawn off and filtered to separate the suspended dirt, and the filtrate collected in a 200 cc distilling

flask. Since fats decompose at about 250C, and on account of the high boiling point of the solvent, it was necessary to conduct the distillation in vaccuo, to eliminate the solvent and recover the fat as residue. The vacuum was obtained by connecting the receiver attached to the condenser, to a May-Nelson Rotary Vacuum Pump (Eimer and Amend, New York, N.Y.) running at 800 R.P.M. The distillation flask was heated over a naked flame and with the vacuum maintained at 21" Hg, the main portion of the solvent was driven over. At this juncture the temperature rose suddenly to about 150-160C, and simultaneously a white solid was observed to be collecting in the condenser tube. This was thought to be the wool fat and the distillation was abruptly stopped, partly to prevent choking of the condenser, and partly because it was considered that the distillation was completed. As the contents of the flask cooled and solidification

began, a marked crystalline formation was observed rather than the amorphous state so characteristic of fats and waxes. This fact aroused some suspicion as to the purity of the fat, and was promptly corroborated on weighing, when it was found that the residue calculated to about 150% on the weight of the original sample of wool. Evidently there remained some constituent of the solvent which had not been eliminated. On further consideration as to the nature of the solvent and a re-examination of the white crystalline solid in the condenser tube, as to physical appearance and odor, the latter was identified as napthalene.

The plan now adopted was to attempt to remove the napthalene by a distillation in steam, and with this end in view, the steam generated in an auxiliary flask was delivered beneath the surface of the molten wool-grease-napthalene liquid and the condensate collected

in a receiver open to the atmosphere. After about one liter of distillate containing most of the contaminating napthalene from the wool grease had been collected, the distillation was stopped in order to drive off, by direct heat, the bulk of water which had condensed in the wool-fat distillation flask and reached a high level thereby threatening, through the violent agitation, to be ejected through the condenser.

During this heating, notwithstanding the caution exercised, a sudden evolution of steam through the heavy layer of floating oil, developed a rapid rise in pressure within the flask, with the result that the stopper was violently ejected from the neck of the flask and most of the contents were forced out.

After this demonstration of difficulties, it was decided to reject the latter portion of the procedure heretofore followed, modifying it by a treatment designed to first eliminate

the troublesome napthalene. This latter treatment depends upon the fact that napthalene is readily sulphonated by concentrated sulphuric acid and the napthalene sulphonate formed, being soluble in the acid is withdrawn along with the "sludge" in the acid layer which separates out below the oil. Hence in the main investigation, which is subsequently to be described, the preliminary step was the refining of the crude oil.

The following figures show the work done on the preliminary treatment with the solvent:

Weight of sample---------- 10 gms.
Solvent used-------------190 cc.
Weight of flask & residue 85.239 gms
Weight of flask---------- 70.475 gms.
Weight of residue-------- 14.764 gms.
% of "wool fat"----------147.64%

CONDUCT OF THE PROCESS.

Refining of Solvent.

The solvent, on its receipt from Chattanooga, was weighed, a sample having first been taken for a subsequent examination. The oil was then drawn off into a ten gallon carboy and concentrated sulphuric acid, amounting to 10% on the weight of original oil was cautiously added, introducing small portions at regular intervals. During this operation, which extended over about seven hours, the liquor was constantly agitated by blowing in air under pressure. The whole mass was allowed to subside over night, and on the following morning the dark oily layer was siphoned off from the acid sludge. The oil was next washed with water to remove the excess of acid, again agitating with air to intensify the washing. After standing four hours to achieve stratification, the oily layer was again withdrawn. To remove the last

traces of acid, a 50% caustic soda solution was added and the whole agitated with air for an hour and allowed to stand over night. On the following morning (3rd day) the oil was separated from the alkaline washings and subsequently submitted to a final washing with water to remove the excess alkali.

The oil, at this stage of its preparation was dark colored and contained suspended matter. It was decided to distil the solvent to a colorless oil, and to obtain for subsequent use in the leaching of the wool, only the light portion which could be easily distilled over.

The distillation was conducted in a steam-jacketed vacuum pan, made by Thomas Burkhard of Brooklyn, New York, and provided with a surface condenser, with receiver connected to a small type of steam driven vacuum pump. The distillation was conducted under 20-23" vacuum, the bulk of solvent distilling over between 200 and 230 F. The temperature was allowed to rise to about 300F and then stopped

because the vapor line leading to the condenser had cooled down sufficiently to indicate that vapors were no longer coming over.

The vacuum was broken by stopping the pump, and the contents of the evaporator were discharged into a suitable receptacle for weighing and examining. This residue was a very thick, heavy, tarry liquid with a peculiarly pungent odor and no longer suitable for solvent purposes and hence, after weighing, was rejected. This served at once to indicate the proportion of the original distillate which was applicable to the process.

When the distillate was withdrawn from the receiver, it was observed that it was contaminated by a large quantity of water. An investigation showed that this was due to a leak in the surface condenser wall between the vapor chamber and the circulating water. Operations were temporarily abandoned for a few days until this fault had been remedied.

In the meantime the oil and water were separated by means of a siphon, the final separation being accomplished in a large separatory funnel.

It was found later that the condenser continued to leak after being fixed, and accordingly, in all of the subsequent distillations it was necessary each time to make this separation of the oil from the water.

The clear oily distillate was now weighed to determine the percent of prepared oil obtained from the refining operations. The following figures show the facts:

Weight of original solvent----- 48.25 lb.
Weight of acid added----------- 4.8 "
Water added to wash------------ 5 litres
Caustic solution added(50%)---- 1½ "
Final wash water added--------- 5 "
Weight of tarry residue-------- 13.13 lb.
Weight of recovered solvent---- 15.75 "
Mechanical losses-------------- 40.1%
Solvent recovered-------------- 32.65%
Tarry Residue------------------ 27.25%

Leaching of the Wool.

Apparatus.

The leaching apparatus consisted of a diffusion battery of five brass-lined, cast iron cells connected by suitable piping as shown in detail in the accompanying blue print. Its operation is explained as follows:

The wool to be leached is packed loosely into the cells through the handholes shown, and when all openings have been tightly closed and secured by clamps, the solvent contained in the monte jus A is forced through the system by admitting air from the pressure tank B, into A upon the surface of the liquid. The air and solvent supply is controlled by the valves shown.

By an examination of the piping, it will be seen that the arrangement of valves is such that all of the cells can be connected in series, and that any cell may be disconnected from the system, for recharging, after its

content of grease has been exhausted by the solvent, without interfering or necessitating a temporary shut down. The process is then continuous.

The design of the apparatus implies that in the leaching operations, as soon as the content of a cell has been leached to the desired extent, it is to be isolated from the system for recharging, and in the ordinary sequence of events, each cell is made to occupy in turn all of the possible positions in the series from first to last. The fresh solvent comes in contact first with the most exhausted wool and is finally driven off from the tap at the bottom of the last digester in the series, which necessarily contains the freshest wool.

Leaching Process.

The weight of wool on hand was one hundred pounds and hence sufficient to provide for a rather extensive leaching process. As

already indicated, however, the volume of solvent resulting from the refining treatment was 15.75 lbs., equivalent to about 2.25 gallons. This volume was just sufficient to fill one of the diffusion cells, and in this contingency it was necessary to abandon the original method of leaching already proposed.

In virtue of this circumstance it was decided to use but three cells in series, eliminating the remaining two from the system by suitable manipulation of the control valves. In addition it was considered that because of the limited supply of oil, the leaching could be accomplished with greater efficiency by allowing the solvent to stand in direct contact with the wool for some time, rather than to depend upon the circulation of the liquid as first planned. Accordingly the battery was charged with wool and the first cell was filled with the solvent and allowed to stand in contact over night, after which time, the

solvent was forced through the other two cells by air pressure from the monte jus, drawing off the liquor from the tap in the bottom of the third cell.

The liquor partially saturated with fat is drained through the perforated copper disc in the bottom of the cell, as a clear fluid, its descent through the wool serving to filter out the mechanically suspended particles. Hence the liquor was taken directly to the evaporator and distilled. The residue consisting of crude grease with a small amount of solvent, was run out and saved. The recovered solvent after siphoning from the water which had leaked into the receiver, was introduced into the second diffuser and allowed to remain in contact with the wool over night. In the morning it was drawn out, allowed to percolate several times through the cell and then distilled, and the residue recovered and united with the first batch obtained. The solvent was

removed from the receiver, siphoned from the water, and placed in the last diffusion cell, and the leaching, distillation, and recovery effected as before.

Preparation of the Wool.

Recovery of Potash.

The moist wool was withdrawn from the diffusion battery, and the excess of solvent removed in a centrifuge, the drainings being returned to the oil already recovered.

The wool was next steeped in warm water for the purpose of dissolving out the potassium salts. After about an hour the water was drawn off and run into a steam-jacketed copper kettle and evaporated under atmospheric pressure. The wool was washed twice more in this manner, the leachings all being united in the evaporator, and the whole concentrated down to a volume of about one gallon. During this process the boiling at one time became so violent, that before it could be brought under control, the liquid rose up over the sides of

the kettle. The loss could not be estimated, yet it was not very great because the liquor was very dilute.

The concentrated potash liquor containing much dirt and suspended impurities was allowed to filter through a bag. This proceeded very slowly and even after three days it was but partly complete, and besides an extremely offensive odor had developed, due to the putrefying organic impurities carried mechanically along from the wool, such as dung. It was thought best to evaporate the liquor to dryness, and to incinerate the residue, thus eliminating the organic matter. The potash was then to be obtained by lixiviating the charred mass with water, followed by filtration and evaporation to a residue.

When the liquor was taken down in the evaporator to dryness, however, the residue resulting was so small as to make the above operations seem undesirable. Accordingly it was

merely weighed and analyzed for potash according to the method subsequently given, so that the percent recovery could be estimated.

Washing of the Wool.

The wool free from potash was now washed in a warm soap solution, an ordinary grade of soap powder being used, to remove adhering dirt. It was then rinsed several times in clear warm water, centrifuged to remove excess water, and dried below 100C.

Nature of the Products.

The Wool.

The wool obtained from the treatment discussed was of light yellow tinge which easily submitted to the bleaching action of either sulphurous acid or a solution of sodium peroxide in hydrochloric acid solution, yielding a fine white wool. Hence the character of the wool was satisfactory. In a practical application of this process it would perhaps be advisable to run the bleach liquor

into the soap solution to accomplish the bleaching and washing in one step.

The Potash.

The consideration involved here is entirely one of yield, and from a comparison based on the analysis of the crude wool and of the residue obtained from the evaporation of the wash water, the recovery is seen to be an efficient one.

The Wool Grease.

As has been stated, the combined residues removed from the vacuum pan contained a small quantity of oil. This was removed by drying the mass in shallow trays in a steam heated vacuum shelf drier under 25"Hg vacuum for two hours. During the drying a part of the fat was lost due to a boiling over of oil in one of the trays. This loss could not be estimated very closely, although roughly it may have amounted to about 25 grams.

The trays were removed from the drier and on cooling their contents solidified,

indicating that, at most, but little solvent remained. The crude wool grease presented in general appearance a brownish yellow to a greenish black color according to the thickness of the layer observed. It was of salve like consistency, and had the characteristic odor of sheep, intensified, due to impurities such as dung and urine products.

Analysis and Results.

Method of Analysis for Wool Grease.

The wool grease, or ether extract, was determined by the familiar Soxhlet extraction method using about a gram sample. Since the potassium soaps are also dissolved by the ether [6], the residue obtained by distilling off the ether was extracted with cold alcohol in which the wool grease is insoluble but the soap dissolves. The alcohol was filtered and evaporated and the residue subtracted from the first residue obtained to give the weight of

[6] Jr.Soc.Chem.Ind. Vol 13, p 1068, W.Herbig

wool grease equivalent to the sample.

Analysis for Potash.

The sample of wool previously leached free from fat with ether, is ignited to burn off the organic matter. The ash is leached with hot water, filtered and the filtrate evaporated to dryness with concentrated HCl to decompose the combined silica. The residue is taken up in hot water and filtered. To the solution add a few drops of nitric acid and a cc. of ferric chloride and a few cc. of ammonium hydroxide. Solution is boiled to precipitate the iron and phosphates and this is filtered off. The solution is evaporated to dryness with sulphuric acid, ignited to expel ammonium salts and weighed as potassium sulphate. This residue may be considered to be potash since the peculiarity of the water soluble portion of the wool is the fact that the potash is almost absolutely free from other metallic oxides.[7] This method is also applicable to

[7] Chemical News Vol. 1, p 203.

the determination of potassium in the potash residue obtained from the evaporation of the leaching waters.

Data on Run.

Wt. of crude solvent----- 48.25 lbs.
Wt. of refined solvent--- 15.75 "
Wt. of wool charged------ 4.125 "
Wt. dry, clean wool------- 3.25 "
Wt. of solvent recovered- 6.62 "
Wt. of crude grease------ .80 "
Wt. of potash residue----- .10 "
% solvent recovered------ 42.00 %
Grease in raw wool------- 29.02 %
Grease recovered--------- 66.89 %
Potash in wool(as K_2SO_4)- 0.85 %
Potash in residue(")-- 37.35 %
Potash recovered---------106.50 %

The potash recovery is practically complete the excess over 100% being due to the fact that the raw wool was weighed out on a scale accurate to one eighth pound while

the analysis was carried out on weights accurate to one thousandth of a gram. Also the weight of potash residue is accurate only to five grams.

Purification of the Wool Grease.

The literature on the subject of "Wool Grease" makes no mention of the purification of the latter, beyond the expression that it is accomplished by "a very mysterious and complex process". On account of lack of time no extensive and systematic investigation for the purification could be attempted. Yet various samples of the crude grease were treated in the manner to be described.

A sample of the crude was agitated with concentrated sulphuric acid which served merely to char and destroy the grease. This might have been expected since this is characteristic of waxes in general.

Other samples were subjected to treatment with dilute sulphuric acid, with caustic soda, and subsequently with such bleaching agents as

sulphurous acid and acid solutions of bichromate and permanganate of potassium.

The best results were obtained by treating twice with 1:3 sulphuric acid, working up the grease continuously with a spatula to hasten the action and to make it uniform. The grease was next worked up in similar manner with sulphurous acid for its bleaching action and subsequently washed with cold water to eliminate the reagents used. Prolonged contact with water must be avoided to prevent the formation of a very stable emulsion (lanoline). The resulting product was of light cream color, with a slight, although not disagreeable odor.

CONCLUSION.

I. The foregoing investigation has demonstrated conclusively that but a part of the solvent proposed is applicable to the process, 32.65% to be exact.

II. The wool by this treatment is readily prepared in a satisfactory form.

III. The process returns valuable by-products in readily utilizable form.

IV. There is open the possibility of reducing the expense involved in the operation of wool washing by virtue of the high return in by-products.

LITERATURE.

The investigation of the literature by the authors revealed no extensive treatise on the subject of "Wool Grease"; but much information which is in some instances of a conflicting nature is to be found in the form

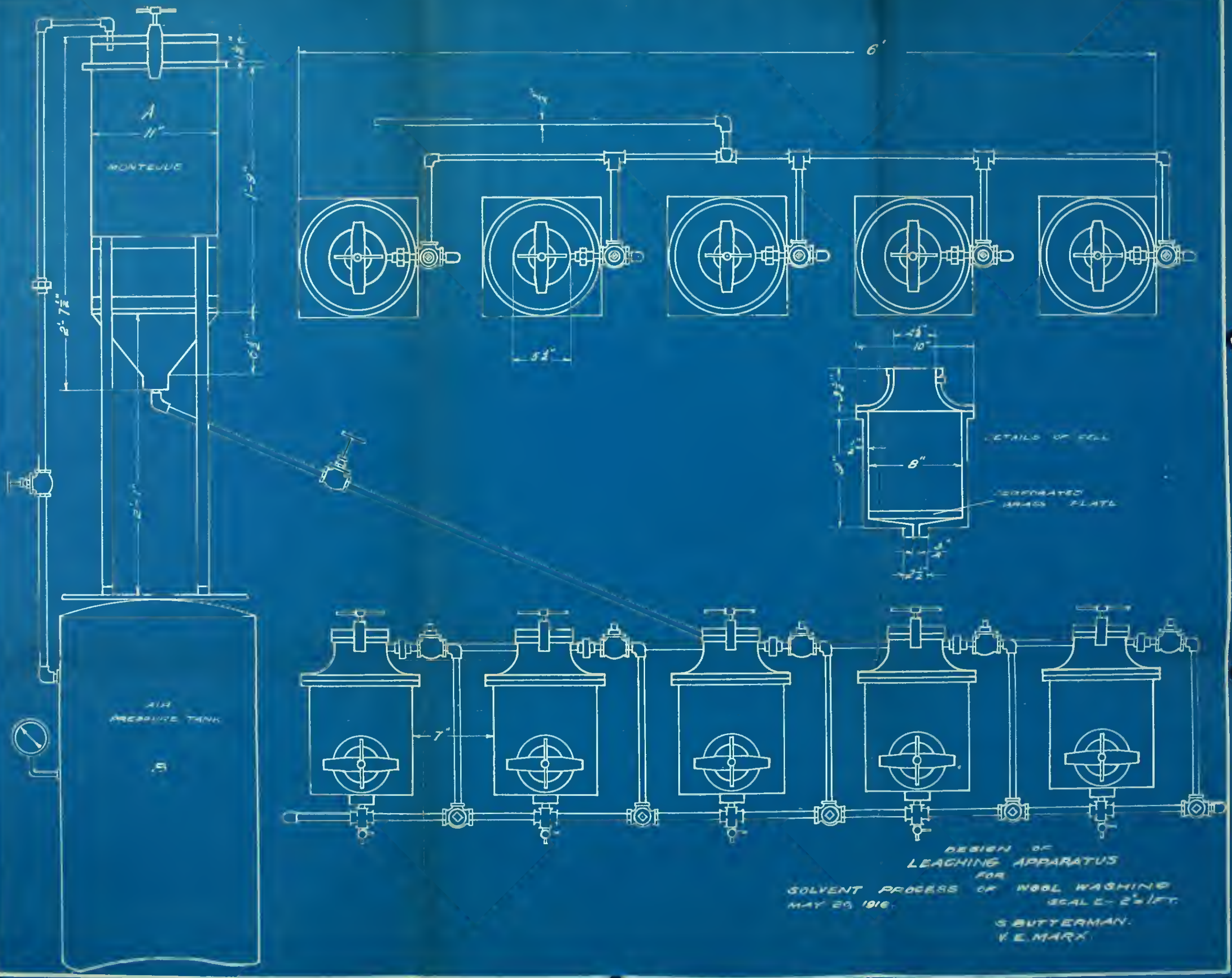
of small articles disseminated throughout the various journals extending many years back, and in modern text books on Industrial Chemistry.

A systematic review was made of the following literature within the range of years indicated below:

Jour. London Chem. Soc.	1883-1907
Chemical News	Vols. 1-100 (complete)
Jour. American Chem. Soc.	1879-1916.
Jour. Soc. Chem. Ind.	1882-1907.
Chemical Abstracts	1907-1916.

The following text books were consulted:

Industrial Chemistry----- Rogers.
Industrial Organic Chemistry- Sadtler.
Industrial Organic Chemistry- Martin
Oils,Fats,and Waxes.--Lewkowitsch.
Commercial Organic Analysis--Allen.



DESIGN OF
LEACHING APPARATUS
FOR
SOLVENT PROCESS OF WOOL WASHING
MAY 29, 1916.
SCALE - 2"=1 FT.

S. BUTTERMAN.
V. E. MARX.